

I. INTRODUCTION

A data quality audit (DQA) was performed on 2018 filter weight data measured using the Office of Research and Development (ORD) National Exposure Research Laboratory (NERL) robotic Measurement Technology Laboratories (MTL) weighing system located in E485A collected under an Office of Air Quality Planning and Standards (OAQPS) Work Assignment (WA) 3-176 on EPA's Research Laboratory Support contract (EP-C-15-008) titled *Mega PE and PM2.5 Round Robin Program Sampling, Gravimetric Analysis, and Distribution* at the request of OAQPS QA Manager, Jenia McBrian. The work is performed by EPA's on-site contractor, Jacobs Technology (Jacobs), Work Assignment Leader (WAL) and balance operator Kyle Digby. Audit activities were performed by Air and Energy Management Division's (AEMD) QA staff Bob Wright and Libby Nessley. Findings are grouped by categories: 1) Balance Performance, and 2) Data Set Analysis & Reporting.

II. AUDIT ACTIVITIES

The following documents and records were provided by OAQPS to complete the DQA:

- Performance Work Statement for RLS WA 3-176
- Sampling Plan for WA 3-176
- Mega PE Filed Operations and Gravimetric Analysis SOPs
- Quality Assurance Guidance Document 2.12-Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods, EPA-454/B-16-001, January 2016.
- Jacobs laboratory research notebook (#4075 issued to Kyle Digby) associated with WA 3-176
- Check weight recertification reports from the EPA NRMRL Metrology Laboratory
- Excel spreadsheets:
 - RR 2018 Raw data_from instrument.xlsx
 - Round_Robin_Fall2018_EPAvsLabs Weights 20190213.xlsx
 - Round_Robin_Fall2018_EPAvsLabs Weights 20190313.xlsx
 - OAQPS-RR-2018-Fall Results_KD_20190326.xlsm
 - 20190516_KD_Fall 2018 Round Robin Master Raw Data Spreadsheet.xlsx
 - 20190516_KD_Maswter_RawData_Fall2018RoundRobinSpreadsheet.xlsx
 - 20190517_KD_MasterResuts_OAQPS-RR-2018-Fall_Jacobs submission.xlsm
 - Corrected OAQPS-RR-2018-Fall Results_4-10-19.xlsm

Auditors reviewed every spreadsheet provided by OAQPS against the raw data from the instrument to determine if there were reporting errors and also performed statistical analyses to determine if there were any balance performance issues with the data sets.

III. AUDIT FINDINGS

Analysis of the balance data indicates that the root cause of the problems related to attaining the 3-microgram acceptance criterion in the weighing protocol (Method 212) lies with the measurements, rather than with the analysis of the data. Specific findings related to balance operation and data set analysis and reporting are detailed below.

BALANCE RELATED

1. Balance calibration and weight certifications are not performed by the same organization.

There is a negative bias where you would expect even distribution on the check weight recertifications performed in EPA's NRMRL Metrology Laboratory. This difference could be related to the fact that the balance is not calibrated by the same organization. Generally, the same organization calibrating the balance should be used to certify the check weights.

2. Check weight certifications are unorganized and inconsistent.

There were numerous certified values for calibration check weights done at various times over the course of the project and it was difficult to keep track of which value should be used in the spreadsheets to determine differences between the actual and obtained values. New weight certifications were also not being updated in the balance software. There is no information in the laboratory research notebook that details when weights were certified and whether or not this information was uploaded to the balance software.

3. Balance stability is not performing up to balance specifications.

Analysis of the balance standard deviations over both the 2017 and the 2018 events indicated the stability of the balance is not performing up to balance specifications. Specifications for the balance are 0.25 µg and the overall average standard deviation for replicate weights was approximately 1.7 µg.

DATA SET ANALYSIS & REPORTING

1. Weigh protocol regarding check weight criteria was not followed.

The Method 212 weigh protocol states that weights for certified check weights must be within <3.1 µg of the certified value for the filter sets bounded by check weights (every 10 filters). If criteria are not met, the filters between the failing check weights must be reweighed. The data sets submitted to EPA by Jacobs as final had multiple instances of differences >3.1 µg without being flagged or reweighing filters. This failure invalidates the data sets unless the issues were documented, discussed with the EPA WACOR, and permission to proceed with modified protocol criteria was given. There are no indications in the laboratory research notebook that protocol criteria were not being met or that a decision to modify the protocol criteria was made.

2. Reported final results from contractor are not consistent with the balance raw data files.

There is evidence that loaded filter weights were mixed up in the file 20190516_KD_Fall 2018 Round Robin Master Raw Data Spreadsheet.xlsx tab labeled <Loaded Weights Preship> compared to the raw data from the instrument tab labeled <loaded fall 1>, specifically for filters T0863045 through T0863080 (see below). This was verified using the weights downloaded directly from the instrument (Column J below/rr2018 raw data.xlsx file). It appears the analyst mixed up the documentation of the filter measurement order, which resulted in some correctly-measured loaded filter weights being incorrectly assigned to other filter IDs. There is evidence the operator noticed the error, attempted to correct the sequences by moving the mixed-up filter weights, but was unable to know with certainty which weights went where (Column O below). It is assumed that for this reason, the post loaded results were carried

through to the final reporting (Column P below) by Jacobs. There was no documentation in the laboratory research notebook regarding a problem with the data set or a filter mix-up. There is also no evidence to determine whether or not this issue was brought to the attention of the EPA WACOR and that the decision to use the post loaded results were discussed and approved.

A	J	K	L	M	N	O	P	Q	R	S	T	U	V	W
OAQPS Filter	Data how it should have been reported						Data how it was reported							
Filter ID	2018 raw data/r loaded fall 1	2018 raw data/r loaded post 1& post 2	2018 raw data/r loaded post 3	2018 raw data/r loaded post 4	2018 raw data/r loaded post 5	20190516_KD_Fall 2018 RR Master Raw Data Spreadsheet/Loaded Weights	20190516_KD_Fall 2018 RR Master Raw Data Spreadsheet/Loaded Weights	20190516_KD_Fall 2018 RR Master Raw Data Spreadsheet/Loaded Weights	20190516_KD_Fall 2018 RR Master Raw Data Spreadsheet/Loaded Weights	2019_KD_Master Results_OAQPS-Fall 2018-2019-Jacobs submission/OAQPS Results	OAQPS-RR-2018-Fall Results_KD_20190326/OAQPS Results	Corrected OAQPS-RR-2018-Fall Results_4-10-19/OAQPS	RR_Fall 2018_EPA AvsLabs Weights 20190213/hip sampled	RR_Fall 2018_EPA AvsLabs Weights 20190313/preshi p exposed
T8636045	380.1814	366.6294				366.6485	366.6294	366.6485	366.6294	366.6485	366.6294	366.6294	366.6294	366.6294
T8636046	376.4213	371.1428				371.1562	371.1428	371.1562	371.1428	371.1562	371.1428	371.1428	371.1428	371.1428
T8636047	372.6187	379.3186				379.3355	379.3186	379.3355	379.3186	379.3355	379.3186	379.3186	379.3186	379.3186
T8636048	372.7015	376.8228	376.8206			376.8376	376.8228	376.8376	376.8228	376.8376	376.8228	376.8228	376.8228	376.8228
T8636049	366.6485	380.1596	360.1546			380.1814	380.1596	380.1814	380.1596	380.1814	380.1596	380.1596	380.1596	380.1596
T8636050	371.1552	376.4000	376.3988			376.4213	376.4000	376.4213	376.4000	376.4213	376.4000	376.4000	376.4000	376.4000
T8636051	379.3355	372.5979	372.5935			372.6187	372.5979	372.6187	372.5979	372.6187	372.5979	372.5979	372.5979	372.5979
T8636052	376.8376	372.6765	372.6750			372.7015	372.6765	372.7015	372.6765	372.7015	372.6765	372.6765	372.6765	372.6765
T8636053	365.4717	365.4520				365.4717	365.4520	365.4717	365.4520	365.4717	365.4717	365.4717	365.4717	365.4717
T8636054	368.0049	367.9878				368.0049	367.9878	368.0049	367.9878	368.0049	367.9878			
T8636055	372.9939	372.9846				372.9939	372.9846	372.9939	372.9846	372.9939	372.9846			
T8636056	367.8445	367.8248				367.8445	367.8248	367.8445	367.8248	367.8445	367.8248			
T8636057	364.6973	364.6801				364.6973	364.6801	364.6973	364.6801	364.6973	364.6801			
T8636058	370.2452	370.2296				370.2452	370.2296	370.2452	370.2296	370.2452	370.2296			
T8636059	372.3346	372.3165	372.3140			372.3346	372.3165	372.3346	372.3165	372.3346	372.3165			
T8636060	369.8722	369.8567				369.8722	369.8567	369.8722	369.8567	369.8722	369.8567			
T8636061	369.3268	369.3092	369.3088			369.3268	369.3092	369.3268	369.3092	369.3268	369.3092			
T8636062	364.0821	364.0688	364.0678			364.0821	364.0688	364.0821	364.0688	364.0821	364.0688			
T8636063	366.8537	366.8331				366.8537	366.8331	366.8537	366.8331	366.8537	366.8331			
T8636064	365.2543	365.2299				365.2543	365.2299	365.2543	365.2299	365.2543	365.2299			
T8636065	366.4282	372.1528	372.1544			372.1522	372.1528	372.1622	372.1528	372.1622	372.1528	372.1528	372.1528	372.1528
T8636066	367.7232	367.7274				369.4094	367.7232	367.7232	367.7232	367.7232	367.7232			
T8636067	366.3451	366.3451				366.7442	366.3451	366.3451	366.3451	366.3451	366.3451			
T8636068	374.4681	367.9004				367.9169	367.9004	367.9169	367.9004	367.9169	367.9004	367.9004	367.9004	367.9004
T8636069	392.0173	368.6519				368.6747	368.6519	368.6747	368.6519	368.6747	368.6519	368.6519	368.6519	368.6519
T8636070	368.4180	369.0473	369.0464	369.0450	369.0452	369.0608	369.0473	369.0608	369.0473	369.0608	369.0473	369.0473	369.0473	369.0473
T8636071	368.8639	369.2411				369.2566	369.2411	369.2566	369.2411	369.2566	369.2411	369.2411	369.2411	369.2411
T8636072	365.3322	372.2397	372.2364			372.2545	372.2397	372.2545	372.2397	372.2545	372.2397	372.2397	372.2397	372.2397
T8636073	372.1622	366.4253	369.5465			366.4282	366.4253	366.4282	366.4253	366.4282	366.4253	366.4253	366.4253	366.4253
T8636074	365.5483	368.5178				368.5257	368.5178	368.5257	368.5178	368.5257	368.5178	368.5178	368.5178	368.5178
T8636075	368.7060	369.5482	369.5486			369.6137	369.5482	369.6137	369.5482	369.6137	369.5482	369.5482	369.5482	369.5482
T8636076	367.9169	374.4564				374.4681	374.4564	374.4681	374.4564	374.4681	374.4564	374.4564	374.4564	374.4564
T8636077	368.6747	391.9989				392.0173	391.9989	392.0173	391.9989	392.0173	391.9989	391.9989	391.9989	391.9989
T8636078	369.0608	368.4124				368.4180	368.4124	368.4180	368.4124	368.4180	368.4124	368.4124	368.4124	368.4124
T8636079	369.2566	366.8380				366.8639	366.8380	366.8639	366.8380	366.8639	366.8380	366.8380	366.8380	366.8380
T8636080	372.2545	365.3188				365.3322	365.3188	365.3322	365.3188	365.3322	365.3188	365.3188	365.3188	365.3188

IV. RECOMMENDATIONS

Based on AEMD's review of the data set and documentation in place for this project the following recommendations should be considered:

- Use the same metrology laboratory (either EPA's or MTL's) to certify the check weights and calibrate the balance to help to eliminate the source of the systematic error.
- To salvage filter data that failed the check weight criteria, use range data to estimate the standard deviation as described in John Keenan Taylor's Statistical Techniques for Data Analysis (Lewi Publishers, 1990). Statistical analysis shows that the precision of the gravimetric measurements is much greater than the 0.25 microgram repeatability that is listed in the protocol criteria. Alternatively, modify the protocol criteria to match balance's actual performance (i.e., 3X standard deviation).
- Service balance on a regular schedule to maintain balance stability such that the 3 µg acceptance criterion can be attained in the future.

- Balance operator needs to document in the laboratory research notebook immediately following each measurement session, whether the acceptance criterion was attained for that measurement session. If not, the measurement session should be repeated.
- Revise SOPs to include quality control steps in the measurement process, specifically with regard to loading the Teflon filters in the automated weighing system's (AWS's) filter carriers and documenting which filters are in which filter carrier.
- Revise QAPP to include additional quality assurance (QA) oversight of both the measurements and the data analysis by both Jacobs and EPA.
- Improve communications among project participants (Jacobs and EPA) so that everyone is aware of any problems that arise during the measurements and data analysis. These communications need to be documented.
- Balance operators should be very careful to properly identify the filters and to measure them in their numeric order. They should carefully document the measurements and data analysis in the project notebook and any data alterations need to be documented in the project notebook
- Jacobs QA manager should be reviewing any measurement data and quality control (QC) check data from QA Category A projects before they are sent to EPA
- EPA Work Assignment Contracting Officer's Representative (WACOR) should be reviewing the measurement data and QC check data when they are received from the contract.